

PATENT SPECIFICATION

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(54) IMPROVEMENTS IN OR RELATING TO APPARATUS FOR GROWING SINGLE CRYSTALS

(71) We, NIPPON ELECTRIC COMPANY, LIMITED, a Company duly organized and existing under the laws of Japan and having its executive office at 7-15, Shiba Gochome, Minato-ku, Tokyo-to, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to an apparatus for growing, by means of a melting process, a single crystal of a material which becomes electrically conductive when heated.

Conventional heating systems for use with an apparatus for growing single crystals by means of a so-called melting process, in which single crystals of semiconductor materials (such as germanium, silicon, gallium arsenide or the like), oxides (such as spinel, garnet, ruby or the like), and metals are obtained from their molten phase, comprise either a high frequency induction heating system or a radiative heating system employing resistive materials. While the high frequency induction heating system involves the possibility of achieving molten phase agitation as a secondary effect, the amount of material that can be melted with such a system is relatively small. For this reason, the resistance heating system is more practical for a large capacity apparatus.

The common single crystal growing apparatus with a resistance heating system brings about virtually no such agitation effect. As a result, the temperature distribution as well as the impurity distribution within the melt are not uniform. This tends to deteriorate the crystallographic characteristics of single crystals produced. To overcome these disadvantages, a technique has heretofore been adopted to rotate the crucible for the melt. This assures, to a certain extent, a uniform impurity distribution in the circumferential direction in the crucible. But in the radial direction, there is no marked improvement at all. Also, it has been difficult to maintain the boundary

between the liquid and solid phases (such boundary will be hereinafter referred to as "interface") flat over a substantial part of the crystal growing period. It is desirable to have a flat interface from the viewpoint of impurity distribution as well as from crystallographic properties. In particular, when the crystal is cut out at right angles to its longitudinal direction, as is done with semiconductor materials such as silicon or germanium, it is essential that the interface be flat.

The present invention consists in an apparatus for growing, by means of a melting process, a single crystal of a material which becomes electrically conductive when heated, the apparatus comprising a heat-resistive container for the material to be melted, and a single means which is disposed around the said container and is supplied, in use, with alternating current which serves to supply both heat and a rotating magnetic field to said material, whereby the material, after melting, is stirred due to the action of the said rotating magnetic field.

Suitably, there is provided a heat-emitting body for heating the material which is constructed in a polyphase configuration (three or more phase) which is supplied with polyphase power. Thus, in one embodiment of the invention, a cylindrical heat-emitting body is arranged outside the crucible. The heat-emitting body is formed with a plurality of slots having axial components to form a plurality of current paths, which are connected in a polyphase configuration, for example, delta-configuration. The heat-emitting body having this delta configuration is supplied with a three-phase current. The arrangement is such that the first phase, second phase, third phase wirings and so on of the supply polyphase alternating current are respectively connected the current paths on the heat-emitting body, arranged around a circuit concentrically with the crucible.

With the above arrangement, the melt within the crucible is subjected to rotation for agitation due to the rotating magnetic field,

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without resorting to any means for physically rotating the crucible itself. The rotation of the melt thus caused is in the circumferential direction in the crucible. Along with this rotation, another sort of rotation, i.e., local rotation, is caused at each of very small regions of the melt. The two different kinds of rotation may be compared the revolution and the rotation of a planet like earth. This electro magnetic agitating technique assures the uniform impurity distribution and flat interface.

The invention will now be described, by way of example, with reference to the accompanying drawings, in which:—

Figure 1 is a schematic sectional view of one embodiment of the invention;

Figure 2 is a plan view of a heat-emitting body used in the embodiment shown in Figure 1;

Figure 3 is a perspective view of the heat-emitting body shown in Figure 2;

Figure 4 is an electrical equivalent circuit of the heat-emitting body shown in Figure 3;

Figure 5 is a block diagram of a power supply system for use with the embodiment of Figure 1;

Figure 6 is a group of curves representing the liquid solid interfaces obtained with a single crystal growing apparatus;

Figure 7 is a perspective view of another heat-emitting body in an apparatus according to the invention; and

Figure 8 shows an electrical equivalent circuit of the heat-emitting body shown in Figure 7.

The apparatus shown in the drawings includes a cylindrical heat-emitting body, which comprises a plurality of current paths equi-angularly spaced apart about the axis thereof. These current paths are connected in a polyphase connection (three phase or more), which is in turn supplied with polyphase power. A material to be melted is disposed centrally within the heat-emitting body, and heated by the current flowing through the current paths. The resulting melt begins to rotate in its crucible due to the rotating magnetic field produced by the current. This renders the temperature distribution uniform throughout the melt and helps facilitate the manufacture of a single crystal of crystallographic excellent properties by pulling from the melt.

Referring to Figure 1, numeral 1 denotes a crucible made of quartz, which is placed within another crucible 2 of carbon, the crucible 2 being mounted on the top of a crucible holding shaft 3. Externally of the crucibles is arranged a hollow cylindrical heat-emitting body 4 in coaxial relationship with the crucibles 1 and 2. The body 4 is externally surrounded by a thermal shield 5 of hollow cylindrical form, disposed co-

axially therewith. All of these components are mounted within a melting chamber 6. A pulling shaft 7 is rotatably and slidably mounted on the top wall of the melting chamber 6 through an aperture formed thereon. A chuck 8 is attached to the lower end of the pulling shaft 7 and is adapted to hold a seed for the growth of a single crystal. Within the crucible 1 is a charge of a material to be melted, that is, the material from which an intended single crystal is to be obtained, such as for example, polycrystalline silicon, to be heated and melted by the heat-emitting body 4. The resulting melt 9 in contact with the seed grows as a single crystal 10 and it grows further as the shaft 7 is pulled upward. An interface is formed between the single crystal 10 and the melt 9.

In the example given, the cylindrical basket-shaped heat-emitting body 4 is formed with slots 12 and 13 extending from one of the end faces 4a and 4b thereof to the extent adjacent the other end, respectively, the slots 12 and 13 being disposed alternately and substantially at equal angular intervals (see Figs. 2 and 3). In addition, three terminals 14, 15 and 16 are integrally formed with the lower end of the cylindrical body 4, substantially at equal angular intervals around the inner periphery thereof. These terminals are formed on the bridging portions for the cut slots 12, which are contiguous with the end face 4b of the heat-emitting body, so as to avoid the short-circuiting of the slots 12 and 13. In this way, resistive current paths 17, 18 and 19 each having a zig-zag form are constituted by the heat-emitting body 4 itself between each adjacent pair of terminals 14, 15 and 16. As viewed from the terminals 14, 15 and 16, the current paths form a three phase delta configuration. Thus, it will be readily understood that using same reference characters for like parts, the electrical equivalent circuit of the heat-emitting body 4 is a delta-connection of resistors 17, 18 and 19 corresponding to the current paths 17, 18 and 19, as shown in Fig. 4.

The heat-emitting body 4 in a three-phase connection is fed with three-phase power. Three electrodes 20, 21 and 22 (22 not shown) extend through apertures formed in the bottom plate of the melting chamber 6, and are vertically aligned with the terminals 14, 15 and 16 and are fixed thereto by bolts 23, for example, for electrical connection between the respective terminals and electrodes and also for mechanically holding the heat-emitting body 4 on these electrodes 20, 21 and 22. To the electrodes 20 and 21, and 22, are respectively connected three-phase wires from a power source not shown.

As shown in Fig. 5, the electrodes 20, 21 and 22 are connected with the secondary side of a three-phase matching transformer 26 which has its primary winding so connected

with a three-phase power supply 28 as to permit the connection of selected two of the three phases to be interchanged by means of a rotational direction control 27. Thus, while one (28a) of the three-phase power supply terminals 28a, 28b and 28c is directly connected with one (35a) of three input lines 35a, 35b and 35c of the matching transformer 26, the terminals 28b and 28c are connected so as to be switchable between the input lines 35b and 35c; and 35c and 35b, by means of switches 36 and 37.

The number and size of slots such as 12 and 13 are chosen to give a desired value of resistance between any pair of terminals 14, 15 and 16, and to give a substantially equal resistance across each of the pairs of terminals. The electrodes 20, 21 and 22 are fixed to the bottom plate of the melting chamber 6 with an insulating material 24 disposed therebetween. A receiving plate 25 is placed on the top of the electrodes 20, 21 and 22, and holds the thermal shield 5 thereon. The heat-emitting body 4 may comprise high purity graphite, for example.

With the apparatus described above, the heat-emitting body 4 is energized by three-phase power to provide heat for melting the material in the crucible 1. The resulting melt within the crucible 1 is then subjected to rotation in a fixed direction.

Experiments have been carried out with twelve slots cut in the heat-emitting body 4 for each of the slots 12 and 13 and connected in the delta-configuration fashion to three-phase power of 50 Hertz. As the crystals began to melt and remaining crystals tended to float on the melt, the latter started rotating slowly in a fixed direction. It was observed that as the melting of the silicon proceeded, the rate of revolution increased. Toward the end of the melting, the rate of revolution of small residual crystals and other residues floating on the melt reached 200 to 300 turns per minute. It was observed that not only the pattern of revolution was concentric, with the center of the crucible as its axis, but it also involved local rotation of parts about their own axes while undergoing the above-mentioned major revolution.

It is evident from these observations that the melt itself is subjected to local rotation, besides the revolution as a whole in the crucible 1. Apparently, both are due to the rotating magnetic field and, particularly, the latter to the eddy current. The rate of the local rotation is higher than the revolution as a whole in the crucible (also as compared with the prior art apparatus in which the crucible was rotated at a rate of several to several tens of turns per minute). Such simultaneous revolution and rotation permit the stirring the melt, uniformizing the temperature distribution in the melt. With the apparatus of the invention, the interface is made

extremely flat (that is, at right angles with respect to the pulling direction) for an extended range of the crystal growth, and the impurity distribution within a plane perpendicular to the direction of pulling is uniform. The latter feature is resulted from the uniform temperature distribution within the melt, brought about by the revolutions of the melt itself.

With a prior art apparatus with a single phase heating, rotation of the crucible is employed, in the course of the crystal pulling. The interface is therefore unavoidably made uneven as shown by curves shown in Fig. 6. Thus, at the initial phase of the crystal growth, the interface is curved to one side of the plane perpendicular to the pulling direction, as indicated by a curve 29. The curvature becomes smaller as the pulling process proceeds to reach a substantially flat state as indicated by curve 31. As the pulling process further proceeds, however, the interface becomes curved to the opposite side, as indicated by curve 30. In contrast, with the apparatus of the invention, while the interface is curved as before at the initial stage of the pulling process, the interface becomes flat after a shorter period of time than with the prior art apparatus, and a subsequent continued pulling maintains the flat condition of the interface. In addition, the prior art apparatus of the kind usually provides a single crystal which includes what is called "core" in the central portion thereof having the impurity distribution different from the peripheral portion. At the boundary between the core and the peripheral portion, the interface unavoidably has uneven portions. However, with the apparatus of the invention, such defects are eliminated, thereby yielding a single crystal of uniform crystallographic and other properties.

Now a quantitative comparison will now be given between single crystals prepared by the prior art single phase heating system and those by the three-phase heating system illustrative of the invention, respectively. The single crystal growing apparatus shown in Fig. 1, that is, the one used in Czochralski process, was used to grow a silicon single crystal in $\langle 111 \rangle$ direction to have a single crystal having an outer diameter of 45 mm and a length of 65 cm. As an impurity, antimony was used at a concentration of 10^{-7} atoms per cubic centimeter.

When the conventional single phase heating system was used, the presence of the core was observed over 70 to 100% of the overall length. With the three-phase heating system, a core was clearly observed immediately below the seed, but gradually faded with an increasing distance from the seed, and eventually disappeared at a distance in excess of 80 mm from the seed.

With the single phase heating system, the

interface varied with the distance from the seed, through a convex form toward the bottom, a flat form and then a convex form toward the pulling shaft. For the three-phase heating system, the interface was convex toward the bottom at a position immediately below the seed, but had a flat form at a distance of 30 mm from the seed and remained flat at further distances.

The resistivity distribution in the diametrical direction was measured by taking the percentage variation:

$$\frac{P_{\max} - P_{\min}}{P_{\min}} \times 100\%$$

where P_{\max} and P_{\min} denote the maximum and minimum values of the resistivity, respectively. With the single phase heating system, the variation ranged from 15 to 30% at a distance of 50 mm from the seed, and ranged from 13 to 20% at a distance of 120 mm. The variation for the three-phase heating system was below 10% at the distance of 50 mm from the seed, and below 8% at the distance of 120 mm.

These figures will be sufficient to show the degree of improvement attributed to the heating arrangement of the invention.

As will be noted from the above description, the three-phase heating system imparts revolutions to the melt in the crucible to provide uniform temperature distribution therein which in turn permits crystallographically excellent single crystals to be obtained. Thus, if desired, the rotating mechanism for the crucible may be abolished, thereby simplifying the entire apparatus. As described above, the control of the switches 36 and 37 of the rotational direction control 27 in the apparatus of the illustrated embodiment to interchange the supply to the two phases of the three-phase feeding system provides a reversal of the direction of rotation of the melt within the crucible. Therefore, it is also possible to obtain further improved single crystals by choosing the direction of rotation of the melt 9 with the direction and the number of revolution of the single crystal pulling shaft 7 as well as the crucible 1 taken into account. The lead wires for the heating power supply each carry $1/\sqrt{3}$ times the current for the single phase heating system, and hence the power consumption can be reduced.

While in the foregoing description, the material used for the heat-emitting body 4 is graphite, it may be replaced by graphite with a silicon carbide coating, or may be composed of tungsten, molybdenum or the like. The terminals 14, 15 and 16 of the heat-emitting body 4 are formed inside the tube-shaped body, but they may be located externally of the body. Furthermore, the slots in the heat-emitting body 4 may be formed helically

rather than rectilinear as illustrated. For example, as shown in Fig. 7, three slots 32, 33 and 34 may extend in parallel with each other from three equally-separated points on the lower end surface 4b of the cylindrical heat-emitting body 4 to points adjacent the upper end surface 4a thereof. Terminals 14, 15 and 16 are formed on the lower end of the body 4 between adjacent slots 32, 33 and 34. In this instance, parts of the body 4 divided by the slots are connected together at the top thereof which remain undivided so that there is formed a star connection as shown in Fig. 8. When a star connection is desired for the heat-emitting body illustrated in Fig. 3, the body may be divided into three parts, with an equal angle subtended by the axis thereof and the parts being electrically connected together at one of their corresponding ends and having their other ends connected to three separate terminals. The power supply may be in a more-than-three phases. In such cases, the number of terminals, and the number of slots or divided parts, if necessary, may be increased or decreased accordingly. Also, while the foregoing description deals with an apparatus using a resistance heater body, the invention can be applied to a polyphase excitation of heater coils of a high frequency induction heating system. With such modification, the uniformity of the temperature distribution of the melt is further improved.

WHAT WE CLAIM IS:—

1. An apparatus for growing, by means of a melting process, a single crystal of a material which becomes electrically conductive when heated, the apparatus comprising a heat-resistive container for the material to be melted, and a single means which is disposed around the said container and is supplied in use with alternating current which serves to apply both heat and a rotating magnetic field to said material, whereby the material, after melting, is stirred due to the action of the said rotating magnetic field.
2. An apparatus as claimed in Claim 1, wherein the said heat and magnetic field applying means has a plurality of current paths connected in at least three phase connection to a source of alternating current.
3. An apparatus as claimed in Claim 2, in which the said heat and magnetic field applying means comprises a cylindrical resistive heat-emitting body, the said heat-emitting body being formed with a plurality of longitudinal slots to form the plurality of the said current paths.
4. An apparatus as claimed in Claim 3, in which the said plurality of longitudinal slots include first slots extending from one end surface of the heat-emitting body to a point adjacent to, but spaced from, the other end surface thereof and second slots extending from the said other end surface to a point

- adjacent to, but spaced from, the said one end face, the said first and second slots being alternately disposed circumferentially of the said body.
- 5 5. An apparatus as claimed in Claim 2, in which the said heat and magnetic field applying means comprises a cylindrical resistive heat-emitting body, the said heat-emitting body being formed with a plurality of substantially parallel slots, the said slots extending in helical form about the axis of the body from one end surface of the body to points adjacent to, but spaced from, the other end face thereof to form the current paths.
- 10 6. An apparatus as claimed in Claim 2, in which the said heat and magnetic field applying means comprises a plurality of high frequency induction heating coils formed in a cylindrical form and is connected to a high frequency power source which has at least three phases. 20
7. An apparatus as claimed in Claim 2, further including means for changing the connection of the current paths to the source, whereby the direction of said stirring effect is reversed. 25
8. An apparatus for growing a single crystal constructed, arranged and adapted to operate substantially as hereinbefore described with reference to, and as illustrated in, the accompanying drawings. 30

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COMPLETE SPECIFICATION

3 SHEETS

This drawing is a reproduction of
the Original on a reduced scale

Sheet 1

FIG. 1.

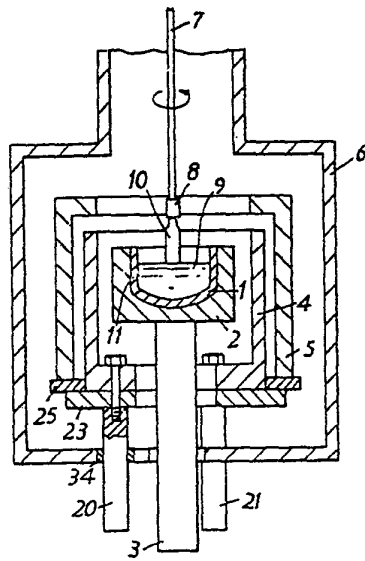


FIG. 2.

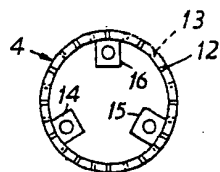


FIG. 3.

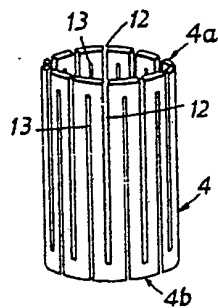


FIG. 4.

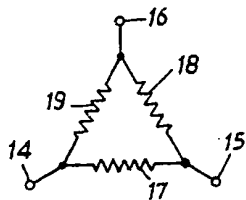


FIG. 5.

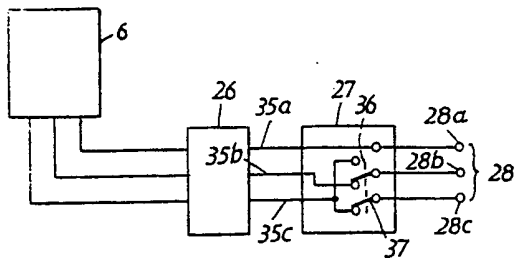


FIG. 6.

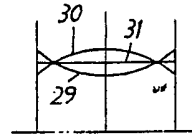


FIG. 7.

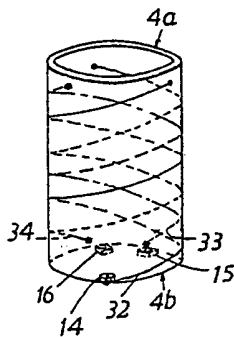


FIG. 8.

